

## Study of the Mineralogical Changes of Clay Due to Cement and Lime Addition Using X Ray Diffraction Technique

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**Abstract:** Light structures in semi arid regions suffer from damage due to expansive subsurface soils subjected to frequent wetting and drying. The use of cement and or lime as stabilizers is a common remedy known to geotechnical engineers. This research is aimed at investigating the mineralogical changes as viewed by x-ray diffraction when lime and cement are added to expansive clay. The x-ray diffraction showed that the mineralogy is not significantly changed due to lime addition while serious alteration and peak collapse of smectite minerals reported with the addition of cement. The percentage of added cement was found proportional to the change in expanding minerals in clay. Adding 6% of cement was found to reduce the expanding minerals of Al-Qatif clay by half compared to 3% cement is added. The stabilization and soil improvement using lime was due to flocculation of clay particles while the improvement obtained by adding cement was due to the cementitious bond and alteration of the mineral structure. Flocculation of clay particles and formation of cementitious bond were found associated with a significant drop in the maximum dry density and changes in the clay compaction characteristics. The outcome of similar studies will serve as a guide to select the appropriate additive and quantity to chemically stabilize clay soils.

**Keywords:** Cement stabilization, lime stabilization, x-ray, minerals

### INTRODUCTION

**General:** Heavily populated semi arid areas are generally covered by large network of roads ranging from express motorways, highways, inter-city artery, ring roads and municipal main or secondary roads. Agricultural links connecting villages and small towns and secondary roads are considered low volume roads. The secondary roads within the cities are designed for low traffic and often consist of a natural sub-grade material ranging from A-2-4 to A-1-b as per AASHTO (2004) classification and overlain by a single 200 mm sub-base layer, 70 mm Asphalt base and 50 mm asphalt wearing course. Thin surfaced pavements which have the lowest standard of any classification are used for agricultural links. These pavements consist of 50 mm of hot mix asphalt spread over top 200 mm granular material classified within A-2-4 group as per AASHTO (2004) classification. The California bearing ratio CBR required is normally 20%.

This type of pavement (secondary or agriculture roads) is very sensitive to subsurface soil conditions. Expansive soil is one of the main factors that causes this type of roads to malfunction. The use of stabilizers such as cement and lime is a known procedure to reduce the expansion caused by swelling soils. This is widely practiced and receiving a wide attention in areas struck by



Fig. 1: A close-up view from a secondary road at Alghatt District



Fig. 2: Distortion and cracks aggravated by seeping water (AL-Zulfi District)



Fig. 3: Damage to Side walks at Umulsahik (Al Qatif area)



Fig. 4: A road subjected to damage and cracks in Tabuk city

expansive soils. Maintenance costs are running into multi million United States dollars annually for a city like Tabuk located to the North-west of Saudi Arabia. Examples of deterioration initiated by expansive soil are shown in Fig. 1, 2, 3 and 4.

Lime and cement are common stabilizers for the subgrade formation. Trial sections are normally suggested in order to observe and assess the improvement added to the road performance. This research was aimed at looking to the effect on the mineralogy when such stabilizers were used. The X-ray diffraction is a widely used technology for the study of minerals.

Smectite is a term used to define a wide range of phyllosilicate minerals. The smectite group of minerals is known of its expanding nature and is generally composed of silica tetrahedral and alumina octahedral units arranged in a three layers order with specific d-spacing separating these units. The chemical composition of each mineral depends on cations present and the way these units are composed. Several forms of expanding minerals were listed in mineral database references. Three of the most common forms are shown as follows :

Montmorillonite:  $(\text{Na,Ca})_{0.33}(\text{Al,Mg})_2\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O}$

This is a whitish, yellow or green clay mineral, composed of tetrahedral and octahedral nets of monoclinic crystal system. Silicates containing aluminum and magnesium and involve sodium and iron.

**Nontronite:**  $\text{Na}_{0.3}\text{Fe}_2(\text{Si,Al})_4\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O}$ . This is a green, olive-green or yellow-green mineral, composed of tetrahedral and octahedral nets of monoclinic crystal system. Silicates containing aluminum and involve sodium and iron.

**Saponite:**  $\text{Ca}_{0.25}(\text{Mg,Fe})_3(\text{Si,Al})_4\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O}$  This is a white, gray-green to bluish, yellow mineral of a greasy or soapy feel appearance composed of tetrahedral and octahedral nets of monoclinic crystal system. Silicates containing aluminum and involve calcium, magnesium and iron.

Montmorillonite is a very soft mineral formed by deposition of volcanic ash in lakes. Montmorillonite, a member of the smectite family, is 2:1 clay, with an average diameter of approximately 1 micrometer. The particle thickness is extremely small (~ 1 nm) (Sharifah, 2006). The water content of montmorillonite is variable. Montmorillonite increases greatly in volume when it absorbs water. Chemically it can be defined as hydrated sodium calcium aluminum magnesium silicate hydroxide. Potassium, iron and other cations are common substitutes and the exact ratio of cations vary with source (Sharifah, 2006). The addition of water causes the montmorillonite to expand to variable levels. However, based on the chemical composition and structure, some types of montmorillonite expand more than other types.

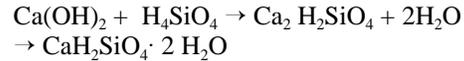
**Lime Treatment:** Lime is generally added to soils to generate long-term strength gain through a series of pozzolanic reactions in the presence of water. The mixture is generally compacted to a desired water content and dry density. The different types of lime available are: quick lime (CaO), hydrated lime (Ca(OH)<sub>2</sub>) and lime slurry which is a suspension of hydrated lime dissolved in water. Quicklime is manufactured by chemically transforming calcium carbonate (limestone-CaCO<sub>3</sub>) into calcium oxide. Hydrated lime is created when quicklime reacts with water. It is the hydrated lime that reacts with clay particles and permanently transforms them into a strong cementitious matrix (National Lime Association, 2004). According to the National Lime Association Publication (Lime-treated Soil Construction Manual, 2004) when lime and water are added to a clayey soil, chemical reactions occur immediately. When the quick lime is added to a soil it will start drying and the moisture holding capacity is then reduced to a great extent. Calcium ions start migrating from the lime to the surface of the clay particles and displace water and weakly bonded anions. The soil becomes friable and starts to granulate and this makes it easily compacted. When lime is added to the soil, the concentration of OH<sup>-</sup> ions increases and elevates the pH level. This will result in the release of alumina and silica into the clay fraction (Ouhadi and Yong, 2003). The released alumina and silica phases react with calcium ions producing CSH (3CaO-2SiO<sub>2</sub>-3H<sub>2</sub>O) and CAH (3CaO-Al<sub>2</sub>

O<sub>3</sub>-Ca(OH)<sub>2</sub>-12H<sub>2</sub>O), which renders cohesive soils workable and less plastic (Raymond and Ouhadi, 2006). In general, the higher the clay contents in soils the higher the plasticity values. Expansive clays have high liquid limits. The addition of lime to highly plastic clay has a significant effect in reducing plasticity index, liquid limit and linear shrinkage and increases shrinkage limit and plastic limit. The compacted soil-lime mixtures have lower maximum density values compared to untreated soil and this reduction increases with the increase in the lime content. The optimum water content also increases proportionally with the increase in the percentage of the lime (Mallela *et al.*, 2004). Lime can modify almost all fine-grained soils, but the noticeable improvement occurs in the case of clayey soils containing minerals such as; smectite, kaolinite and illite. The soil mineralogy is an important issue for the effectiveness of lime treatment. Generally, the optimum amount of lime required for soil treatment is usually between 2 and 8 % measured by dry weight of the soil (Basma and Tuncer, 1991).

**Cement Treatment:** Cement is a comprehensive chemical stabilizer often used in ground improvement. The most common cement type used to treat clayey soils is the Portland cement which when combined with water, hydrates to form the cementing compounds of calcium-silicate-hydrate and calcium-aluminates-hydrate, as well as excess calcium hydroxide. Calcium hydroxide released during the process of hydration reacts with soil alumina and soil silica fine-grained clay soils. This pozzolanic reaction is an important aspect of the stabilization of clayey soils. Portland cement essentially contains five main components. In their pure form, these phases are tricalcium silicate (Ca<sub>3</sub>SiO<sub>5</sub> or C<sub>3</sub>S) (Taylor, 1997), dicalcium silicate (Ca<sub>2</sub>SiO<sub>4</sub> or C<sub>2</sub>S), tricalcium aluminate (Ca<sub>3</sub>Al<sub>2</sub>O<sub>6</sub> or C<sub>3</sub>A), a tetracalcium aluminato ferrite (often given as Ca<sub>4</sub>Al<sub>2</sub>Fe<sub>2</sub>O<sub>10</sub> or C<sub>4</sub>AF) and a sulfate phase (often gypsum, CaSO<sub>4</sub>·2H<sub>2</sub>O or CSH<sub>2</sub>) (Wesselsky and Jensen, 2009).

Cement plays an important role in altering and enhancing many physical properties of the soil such as; plasticity, compaction, durability, compressibility, swell potential and shear strength.

**Pozzolanic reaction and its compounds:** The Pozzolanic reaction is a chemical reaction which occurs in cement or in a mixture of hydrated lime (calcium hydroxide) with amorphous siliceous materials in the presence of water. The most important reaction of the siliceous pozzolans occurs between reactive silica, lime and water to produce calcium silicate hydrate (Helmuth, 1987). A simple acid-base reaction between Calcium Hydroxide, (Ca(OH)<sub>2</sub>) and silicic acid, (H<sub>4</sub>SiO<sub>4</sub>, or Si(OH)<sub>4</sub>), can be shown as an example of pozzolanic reactions. According to Cook (1986), the reaction can be schematically represented as follows:



The above reaction can be simplified into:



The product of (CaH<sub>2</sub>SiO<sub>4</sub>·2H<sub>2</sub>O) formed is a calcium silicate hydrate, also abbreviated as CSH in cement chemist notation (Cook, 1986).

Hydration reaction is the main hardening reaction of calcium silicate binders due to the reaction of calcium silicates, calcium aluminates and calcium-silicate-aluminate phases (i.e., 2CaO·Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub> and 4CaO·Al<sub>2</sub>O<sub>3</sub>·Fe<sub>2</sub>O<sub>3</sub>) with water (Cizer *et al.*, 2009). This leads to the formation of hydrated compounds such as calcium silicate hydrates (CSH), calcium aluminate hydrates (CAH) and AFm phases (AFm phase is an alumina and monosulfate phase) (Taylor, 1997).

Calcium silicate hydrates possess a remarkable level of structural complexity (Chena *et al.*, 2004). More than 30 crystalline calcium silicate hydrate phases are known, these phases range from semicrystalline to nearly amorphous, all of which are described by the generic term, “C-S-H” (Taylor, 1997). The C-S-H formed by the hydration of tricalcium silicate (C<sub>3</sub>S) or β-dicalcium silicate (β-C<sub>2</sub>S), which together constitute about 75 % of a portland cement by weight, is particularly a disordered variety, usually called C-S-H gel to distinguish it from C-S-H phases made by “synthetic” preparation routes such as the reaction of CaO and SiO<sub>2</sub> or the double decomposition of a Ca salt and an alkali silicate in aqueous solution (Chena *et al.*, 2004). CSH has many crystal structures, some of which are depicted by Richardson (2008) as; Foshagite Ca<sub>4</sub>(Si<sub>3</sub>O<sub>9</sub>)(OH)<sub>2</sub>, Nekoite Ca<sub>3</sub>Si<sub>6</sub>O<sub>15</sub>·7H<sub>2</sub>O, Clinotobermorite Ca<sub>5</sub>Si<sub>6</sub>O<sub>17</sub>·5H<sub>2</sub>O, Tobermorite Ca<sub>5</sub>Si<sub>6</sub>O<sub>16</sub>(OH)<sub>2</sub> and Jennite Ca<sub>9</sub>Si<sub>6</sub>O<sub>18</sub>(OH)<sub>2</sub>·8H<sub>2</sub>O.

Calcium aluminate hydrates are formed when Ca<sub>3</sub>Al<sub>2</sub>O<sub>6</sub>, a compound present in Portland cement, reacts with water (Spierings and Stein, 1978). The chemical formulation of Calcium Aluminate Hydrate (CAH) is (3CaO·Al<sub>2</sub>O<sub>3</sub>·Ca(OH)<sub>2</sub>·12H<sub>2</sub>O). CAH has many crystal structures. Richardson (2008) depicted some of the crystal structures of CAH as; Bicchulite Ca<sub>2</sub>(Al<sub>2</sub>SiO<sub>2</sub>)(OH)<sub>2</sub>, Katoite Hydrogarnet Ca<sub>1.46</sub>AlSi<sub>0.55</sub>O<sub>6</sub>H<sub>3.78</sub> and Strätlingite Ca<sub>2</sub>Al(AlSi)<sub>2.22</sub>O<sub>2</sub>(OH)<sub>122.25</sub>H<sub>2</sub>O.

**Detecting CSH and CAH Phases:** This study is aimed at utilizing the x-ray diffraction technique to view the possible changes in the minerals and the new forms of compounds formed as a result of adding lime or cement. During the application of cement or lime to soil, peaks

pertaining to cementing compounds such as CSH and CAH were observed. According to Al-Mukhtar et al. (2010) new peaks corresponding to calcium aluminate hydrate (CAH) were formed  $\text{Ca}_3\text{Al}_2\text{O}_6 \times \text{H}_2\text{O}$  appeared at d-spacing of 0.76, 0.376, 0.285 and 0.166 nm at  $2\theta = 11.6^\circ, 23.6^\circ, 31.3^\circ$  and  $55.3^\circ$ ; respectively in all the XRD patterns of treated sample. Two new peaks appeared at d-spacing of 0.308 and 0.279 nm at  $2\theta = 29.0^\circ$  and  $32.1^\circ$ . These peaks correspond to calcium silicate hydrate (CSH)  $\text{Ca}_3\text{Si}_2\text{O}_7 \times \text{H}_2\text{O}$ . The size and pattern of x-ray peaks of the expanding minerals in pure clay can be compared to clay treated with lime and cement.

## MATERIALS AND METHODS

**Material and Equipment:** This work was carried out during the years 2010 and 2011 at King Saud University in Saudi Arabia. The soil material selected for this study was brought from Al-Qatif district from the eastern province of Saudi Arabia. The physical and geotechnical properties of Al Qatif clay is given in the Table 1:

The x-ray diffraction was carried out using SHIMADZU XRD-7000 system at 40 Kv Cu K alpha radiation source and a scanning speed of 2 degrees/minute. Divergence and scatter slit is 1 degree. Receiving slit is 0.3 mm. Continuous scanning for two theta range of 2 to 40 degrees was considered.

Sample preparation is an essential parameter that governs the intensity measurements and can be a basic factor in the quantitative analysis (Rietveld, 1969). Several other parameters can affect the intensity measurements; these include instrument related parameters (e.g., Variation in power supply, temperature and diffractometer efficiency). The intensity of reflection is also dependant on the mass absorption coefficient of the tested material. In this study sample preparation was kept identical for all tested samples and same machine was utilized with the same set up conditions applied for all tests. Air dried samples of crushed material passing sieve number 200 (size 75  $\mu\text{m}$ ) were used.

**Testing Program:** The x-ray diffraction technique is generally used in qualitative and quantitative mineralogical identification. The x-radiation is produced when high energy cathode rays rip electrons from a target element placed in the anode of an evacuated tube. The wave length produced is short and is dependent on the target material. The radiation involves two components known as  $k\alpha$  and  $k\beta$ . Screens were used to filter  $k\beta$  for simplification. Crystal spacing can be determined by x-rays of a single wave length. The common radiations used in x-ray crystallography are produced by Cu, Co, Fe and Cr tubes. Powder prepared samples from Al-Qatif were tested.

Table 1: Physical Properties of Al-Qatif soil

Liquid Limit (%)	136
Plastic Limit (%)	60
Shrinkage Limit (%)	12
Linear Shrinkage (%)	28
Plasticity Index (%)	76
Specific Gravity	2.70
% finer than 200 $\mu\text{m}$	99.1
USCS Classification	CH

The basal spacing at peaks in the range of  $d = 12 \text{ \AA}$  to  $15 \text{ \AA}$  indicates the presence of smectite minerals.  $\text{Na}^+$ -montmorillonite d spacing is  $12 \text{ \AA}$  while  $\text{Ca}^{++}$ -montmorillonite is 14 to  $15 \text{ \AA}$ . Other minerals including Vermiculite and Chlorite can also occur at  $d = 14 \text{ \AA}$ . The Illite can be observed at  $d = 10 \text{ \AA}$ . From the X-ray diffraction results it is possible to confirm the presence of expanding minerals belonging to smectite groups. Some techniques are commonly used to differentiate between overlapping peaks including glycolation, heating and other means. Some peaks shift or disappear when a special treatment is used. This study used Al Qatif clay, a typical semi-arid highly expansive material. Peak intensities can tell about mineral proportions when a calibrated curve is prepared for different concentrations.

The test program was planned as to have the x-ray diffraction profile for Al-Qatif clay prepared at room temperature after saturating with potassium chloride. The aim of chemical saturations was to characterize different clay minerals including smectite, illite and kaolinite. Heating the sample to  $550^\circ\text{C}$  was adopted to detect the changes in d-spacing and collapse of expanding mineral peaks. The peak intensity changes were observed. Samples saturated with potassium chloride but including cement and lime as additives were investigated. The ranges tested included 3% cement, 6% cement, 4% lime and 8% lime. All preparation procedures and testing conditions were kept similar in order to enable good comparison. 1 M potassium chloride reagent was used for air dried slides.

As part of this study additional physical testing was carried out to compare the changes in compaction characteristics. One of the measures for the stabilization in clays is the compaction test and compaction characteristics. These tests describe the relationship between the moisture content and the dry density. Changes in the curve showing this relationship due to the addition of lime or cement were observed.

The compaction tests were carried out using the modified proctor approach as given in ASTM D1557. The test was carried out on pure clay and on clay treated with 4% lime, 8% lime, 3% cement and 6% cement. The moisture content and the dry density plot were obtained. Maximum dry density and optimum moisture content was defined for each sample. OPM is the optimum moisture content at which maximum dry density occurs. WOPM refers to the wet side of the curve or values of dry density when moisture content is greater than the optimum.

Table 2: Peak areas and intensities of Al Qatif Clay compared to lime and cement clay mixtures

Prepared sample	Reference area	Reference peak	Area under the curve for 2θ	Peak intensity for 2θ = 2 to 10°	Area increase (%)	Peak increase (%)
Pure Clay saturated in KCL	41	1372	41	3172	0	0
Clay with 3% cement saturated in KCL	41	1372	30	2604	27.5	42.0
Clay with 6% cement saturated in KCL	41	1372	17	1764	58.5	100
Clay with 4% lime saturated in KCL	41	1372	41	3530	0	0
Clay with 8% lime saturated in KCL	41	1372	36	2900	12.2	19.3

Al-Qatif clay was prepared at room temperature after saturating with potassium chloride. Heating the samples to 550°C was adopted to detect the changes in x-ray profile and d-spacing. Samples saturated with potassium chloride but including cement and lime as additives were investigated using the experimental system as described above.

### RESULTS AND DISCUSSION

The swelling and volume change behavior is dependant on the clay minerals present in a soil. Adding cement or lime will affect the chemical balance and may lead to some changes in the structure of minerals or even alter or convert the mineral. Evidence of this can be detected by the use of x-ray diffraction technique. As part of this research x-ray diffraction was carried out utilizing SHIMADZU XRD-7000 system at 40 Kv Cu K alpha radiation source and a scanning speed of 2 degrees/minute.

The presence of the smectite clay minerals, illite and kaolinite was confirmed for Al Qatif pure clay sample saturated with potassium chloride. On glycolation the d-spacing was increased to 18 Å. On heating to 550°C most of the smectite minerals peaks collapsed and a maximum d spacing of 10 Å was reported. Non-clay minerals including quartz, feldspar and calcite were also reported. The d-spacing for illite was around 9.9-10.1 Å and it remained constant with MgCl, Ethylene Glycol and KCl saturations. The d-spacing of kaolinite was 7.15 Å with MgCl, Ethylene Glycol and KCl saturations and the kaolinite peaks disappeared when heated at 550 degrees.

The intention was to see how the addition of lime (4% and 8% by weight) or cement (3 and 6% by weight) will affect the mineralogy of the swelling clay of Al Qatif. The comparison was made between the diffraction profiles of the clay-cement mixtures, clay-lime mixtures and the natural Qatif clay soaked in Potassium Chloride (KCl) at room temperature and when heated to 550 degrees. The heating cause's smectite group peaks to collapse and expanding minerals to disappear. Works of Al-Rawas *et al.* (2005) showed that montmorillonite, which is the main mineral responsible for swelling, collapsed on heating beyond 550 degrees and the swelling was reduced to zero. The laboratory test results of Al-Rawas *et al.* (2005) indicated that the swell potential and the swelling pressure converged to zero upon elevated

heating (800°C). Basal spacing of smectite group is sensitive to interlayer hydration. Dehydrated montmorillonite may have a 10 to 13.6 Angstrom spacing and can move up to 18 Angstrom on full hydration. Mordome *et al.* (2009) compared the response of basal spacing of Ca montmorillonite and Na montmorillonite and found that differences are related to hydration powers of exchangeable cations. The peak intensity is proportional to the mineral content in the sample and measurements of peaks can provide good information for minerals quantitative analysis. It is known that varying absorption coefficient for different mineral phases in a mixture can affect the peak intensity (Al-Jaroudi *et al.*, 2007).

One of the approaches of quantitative analysis of minerals is based on comparing the peak size or the area under the peak of the x-ray diffraction profile to that of known standard mineral concentration. The quantitative analysis for specific mineral using internal or external standard method (Dafalla and Ali, 1991) refers to the area under the peak that match the area calibrated using a known mineral. The amount of the smectite group minerals content reflected by these measures can tell about how much minerals are likely altered or collapsed. In this study the areas were measured for comparison in order to check if significant part of the smectite group minerals is altered. The percentage change in areas or intensity do not give the percentage of minerals altered but can confirm the order by which change is taking place. Two new terms are used here to define a base line; these are reference area and reference peak. The reference area is the area under the diffraction profile for the zone (two theta = 2 to 10 degrees) subtracted by the area under the pure clay diffraction profile heated to 550°C for the same zone. The reference peak is the peak height measured for the pure clay subtracted by the peak height for the heated pure clay. Table 2 summarizes the peak heights and relative areas as measured for the tested samples.

The reactions between lime and the clay include exchange of cations, flocculation, carbonation and pozzolanic reactions (Al-Rawas *et al.* 2005). The cation exchange causes clay particles to flocculate or getting close to each other leading to the so called flocculation. Flocculation is responsible for modification of engineering properties. In the case of adding cement, reactions due to calcium silicate and aluminates hydration gives cementitious bond within the soil particles or alter the mineral composition and/or structure.

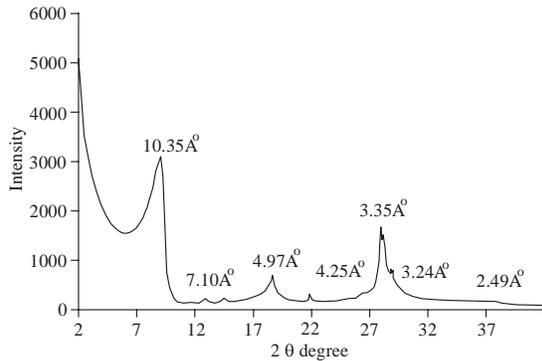


Fig. 5: X- Ray profile for pure clay treated with potassium chloride

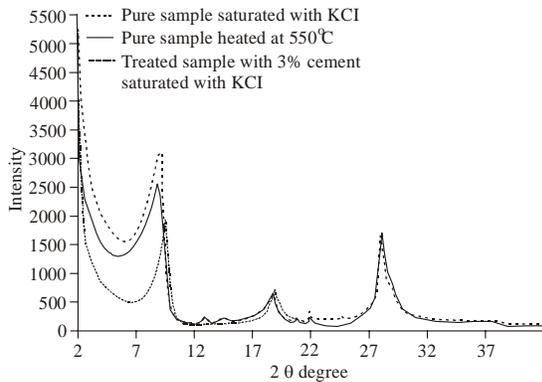


Fig. 6: X-ray profile for 3% cement clay mixture compared to pure AlQatif Clay (heated to 550° and non-heated)

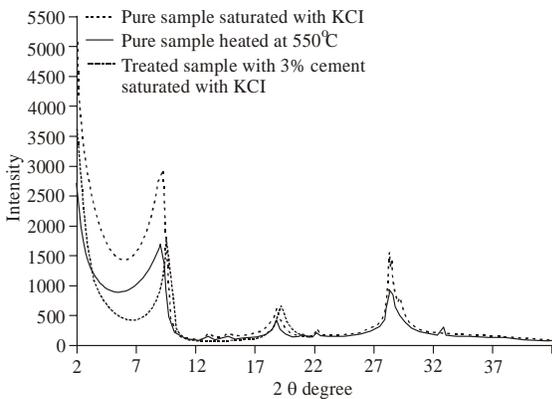


Fig. 7: X-ray profile for 6% cement clay mixture compared to pure AlQatif Clay (heated to 550° and non- heated)

The outcome of this x ray study indicates that adding of cement can alter some smectite minerals and reduce their expansion nature through a cementitious bond while when adding lime the expansion is reduced due to flocculation of clay particles and the mineralogy is not significantly altered. From this study we note that improvement using cement was proportional to the

Table 3: The effect of the lime and cement on compaction properties of Al-Qatif soil

Properties	4% lime	8% lime	3% cement	6% cement
Increase in OMC	3.1	6.2	0	25
Increase in WOMC	13.9	16.7	13.9	30.6
Decrease in Dry	4.8	9.4	4.2	5.2
Density at OMC				
Decrease in Dry	4.9	9.9	4.4	5.6
Density at WOMC				

\* Values given are percentage change based on untreated soil.

quantity added i.e., the quantity of added cement is a function of the level of improvement. Area increase or peak increase was not observed when adding 4% of lime. This amount may not be sufficient to produce flocculation or hold the expansion and tension stresses. The 8% addition of lime caused an increase in the area under peak and peak intensity by 12.2 and 19.3% respectively.

The results in Table 3 indicated that adding lime or cement shifts the moisture density curve to the right and increases the optimum moisture content for both lime and cement. The other clear change was the decrease in the dry density at the optimum moisture content and also wet of optimum moisture content. This reduction is attributed to the formation of lumps and aggregations of clay particles which are not breakable at the level of compaction energy commonly used in standard and conventional compaction tests.

The flocculation or aggregation caused by adding lime is not of sufficient bond to hold the clay against tensile stress. The clay-lime mixture may be friable and the improvement is function of the amount added. The influence of the pozzolanic reactions is not satisfactory to cause significant changes in the structure of the expanding minerals. The amount of lime additive that can stabilize swelling clays depends on the type of clay and its chemical composition.

From this XRD study it was indicated that, lime has an effect in changing the mineralogy of Al-Qatif soil by changing the diffraction profile at 8% and forming pozzolanic compounds in the form of Calcium Aluminate Hydrate (CAH) at  $2\theta = 31$  degrees. Similar outcome is reported by Al-Mukhtar *et al.* (2010). These cementing agents are further responsible for the improved soil strength and swelling reduction resulting from the stabilization process (Mohamed and Al-Hosani, 2000).

To evaluate the effect of cement treatment on clay mineralogy, a similar approach and concept as described in the experimental section below was used. From Fig. 6 and 7 and Table 3 for Al-Qatif soil, it is inferred that the area under the curve and the peak intensity from  $2\theta = 2$  to  $2\theta = 10$  degrees decreased on the addition of 3% and 6% cement. The reduction was more pronounced in the case of 6% cement. The outcome of this XRD study indicates that, the amount of expanding minerals were significantly reduced and the cement had an effect in changing the mineralogy of the soil as a result of pozzolanic reactions

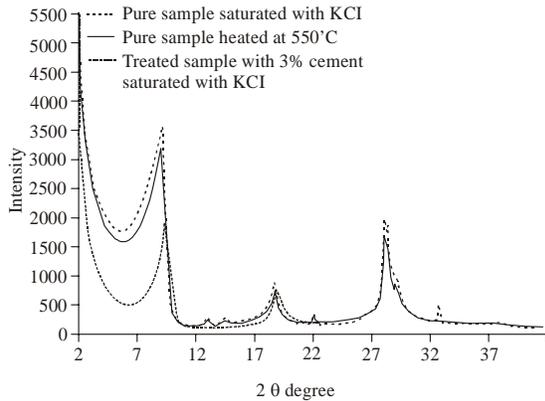


Fig. 8: X-ray profile for 4% lime clay mixture compared to pure AlQatif Clay (heated to 550° and non-heated)

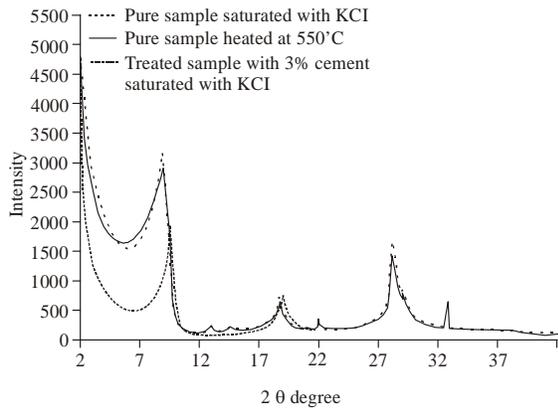


Fig. 9: X-ray profile for 8% lime clay mixture compared to pure AlQatif Clay (heated to 550° and non-heated)

taking place. A strong bond was generally created which prevented the expanding minerals from causing high expansion. These cementitious compounds were responsible for the remarkable reduction in the swelling for Al-Qatif soil.

It can be concluded that by adding cement to clayey soils, the pozzolanic reactions can cause some minerals to change and form other cementitious compounds and the original concentration of expanding minerals within the clay will reduce.

Figure 8 and 9 presents the XRD profiles for the 4% and 8% lime treated clay respectively. As discussed above the influence of the pozzolanic reactions is not strong enough to cause significant mineral structure changes when considering low lime concentrations.

The compaction characteristics of lime-treated and cement-treated clays are presented in Fig. 10 and 11. The optimum dry unit weight drops significantly with the increase of lime concentrations.

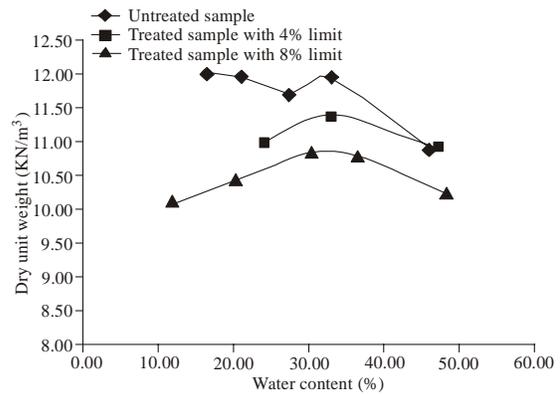


Fig. 10: Compaction Characteristics of Lime-treated Al-Qatif Clay

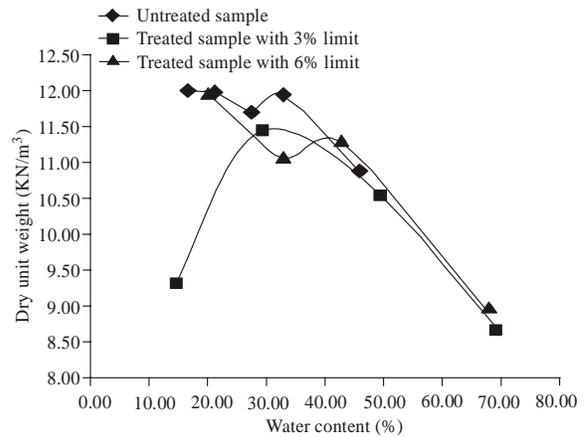


Fig. 11: Compaction Characteristics of Cement-treated Al-Qatif Clay

### CONCLUSION

Based on the works carried out and test results reported in this research, it can be concluded that the addition of cement to a potentially expansive clay can alter some smectite minerals and can reduce their expansion nature through forming cementitious bonds. The addition of lime to potentially expansive clay can reduce the expansion due to flocculation of clay particles and the mineralogy is not significantly altered. The change and decrease in expanding minerals when using cement is proportional to the quantity added. When adding 6% quantity of cement to a clay, reduction in area under the peak will be twice that produced by a clay with 3% cement added. The pozzolanic reactions associated with lime are of weaker influence in altering the structure of expanding minerals. Flocculation of clay particles and formation of cementitious bond were found associated with a drop in the maximum dry density of the clay and significant changes in the clay compaction characteristics.

### ABBREVIATIONS

AASHTO American Association of State Highway and Transportation Officials  
CBR The California Bearing Ratio  
CH Highly plastic clay  
CSH (3CaO-2SiO<sub>2</sub>-3H<sub>2</sub>O) calcium silicate hydrates  
CAH (3CaO-Al<sub>2</sub>O<sub>3</sub>-Ca(OH)<sub>2</sub>-12H<sub>2</sub>O) calcium aluminate hydrates  
KCL potassium chloride  
OMC optimum moisture content  
WOMC wet of optimum moisture content  
°A Angstrom  
°C degree in Centigrade  
Al Aluminum  
C Carbon  
Cu Cupper  
Ca Calcium  
Fe Iron  
H Hydrogen  
Mg Magnisium  
Na Sodium  
Si Silicon  
O Oxygen  
k $\alpha$  Radiation component  
k $\beta$  Radiation component

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### REFERENCES

AASHTO, 2004 Standards. American Association of State Highway and Transportation Officials, M-145-191.  
Sharifah, N.S.J., 2006. Adsorption Study-Dye Removal Using Clay. A report in partial fulfillment of the requirements for the award of the degree of Bachelor of Chemical Engineering. Faculty of Chemical Engineering and Natural Resources, University College of Engineering and Technology Malaysia.  
National Lime Association, 2004. Lime-treated soil construction manual. Lime stabilization and lime modification. Published by National Lime Association, January 2004.  
Ouhadi and Yong, 2003. The role of clay fraction of marly soils on their post stabilization failure. Eng. Geol., 70: 365-375.

Raymond, N.Y. and V.R. Ouhadi, 2006. Experimental study on instability of bases on natural and lime/cement stabilized clayey soils. Appl. Clay Sci., 35(3-4): 238-249.  
Mallela, J., H.V. Quintus and K. Smith, 2004. Consideration of lime-stabilized layers in mechanistic-empirical pavement design. The National Lime Association.  
Basma, AA. and E.R. Tuncer, 1991. Effect of lime on volume change and compressibility of expansive clays. Transportation Research Board, Washington DC., TRR No. 1296, p: 54-61.  
Taylor, H.F.W., 1997. Cement Chemistry. 2nd Edn., Thomas Telford, London.  
Wesselsky, A. and O.M. Jensen, 2009. Synthesis of pure Portland cement phases. Cement Concrete Res., 39: 973-980.  
Helmuth, R., 1987. Fly Ash in Cement and Concrete. Portland Cement Association, Skokie, Ill., p: 203.  
Cook, D.J., 1986. Natural pozzolanas. In: Swamy R.N., Editor Cement Replacement Materials, Surrey University Press, p: 200.  
Cizer, O., K. Van Balen, D. Van Gemert and J. Elsen, 2009. Competition between carbonation and hydration on the hardening of calcium hydroxide and calcium silicate binders. In: Building Materials and Building Technology to preserve the Built Heritage, 2(33-2), (Schueremans, L. (Eds.)). 1st WTA-International PhD Symposium "Building Materials and Building Technology to preserve the Built Heritage". Leuven, Belgium, 8-9 October 2009 Karlsruhe, Germany: WTA Publications. pp: 353-368.  
Chena, J., J. Thomas, H.F.W. Taylor and H.M. Jennings, 2004. Solubility and structure of calcium silicate hydrate. Cement Concrete Res., 34: 1499-1519.  
Richardson, I.G., 2008. The calcium silicate hydrates. Cement Concrete Res., 38: 137-158.  
Spierings, G.A.C.M. and H.N. Stein, 1978. Electrokinetic properties of calcium alnminate hydrates. Colloid Polym. Sci.; 256: 369-374.  
Al-Mukhtar, M., L. Abdelmadjid and F.A. Jean, 2010. Behaviour and mineralogy changes in lime-treated expansive soil at 50° C. Appl. Clay Sci. J., 50: 199-203.  
Rietveld, H.M., 1969. A profile refinement method for nuclear and magnetic structures. J. Appl. Crystallograp., 2: 65-71.  
ASTM American Standard Testing Methods, ASTM D 1557.  
Al-Rawas, A.A., A.W. Hago and H. Al-Sarmi, 2005. Effect of lime, cement and Sarooj (artificial pozzolan) on the swelling potential of an expansive soil from Oman. Build. Environ., 40(5).

- Mordome, S.K. and Katsuyki, 2009. Swelling behaviour of Na-and Ca-Montmorillonite up to 150° C by insitu X-ray Diffraction Experiments. *Clays Clay Miner.*, 57(2): 150-160(11).
- Al-Jaroudi, S.S., A. Ul Humaid, A. Mohammed and S. Saner, 2007. Use of X-ray powder diffraction for quantitative analysis of carbonate rock reservoir samples. *Powder Techn.*, 175: 115-121.
- Dafalla, M.A. and E.M. Ali, 1991. Quantitative identification of montmorillonite in clays using X-ray diffraction'. *Australian Geomechanics*.
- Mohamed, A.M.O. and K.I. Al-Hosani, 2000. Waste utilization from cement industry for modification of soil properties in Physicoarid lands. *Int. Conf. Geoenviron. Muscat. Oman*, pp: 1-12.